

METHOD FO MANUFACTURE OF ALUMINIUM ALLOY SHEETS CONTAININGMAGNESIUM AND ZINE

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The present invention resides in the manufacture of an AlMg alloy sheet with zinc addition which is readily deformable in the soft condition and especially suited for shaping into motor vehicle body components. The alloy sheet has a strength of at least 250 N/mm², a grain size of less than 50 μ m and, after deformation has occurred, remains free of surface defects such as orange peel effect and Luders bands and also, after a possible influence of heat, remains insensitive to stress corrosion cracking. The zinc addition produces a widening of the working range so that after the last cold rolling operation the alloy can be soft annealed above the recrystallization temperature without coarse grains or Luders band appearing. The zinc addition produces an insensitivity with respect to stress corrosion cracking after a heterogenization annealing subsequent to the soft annealing, even after a sensitization at 150 DEG C. on a sheet which has been deformed with a cold reduction of 20% and more.

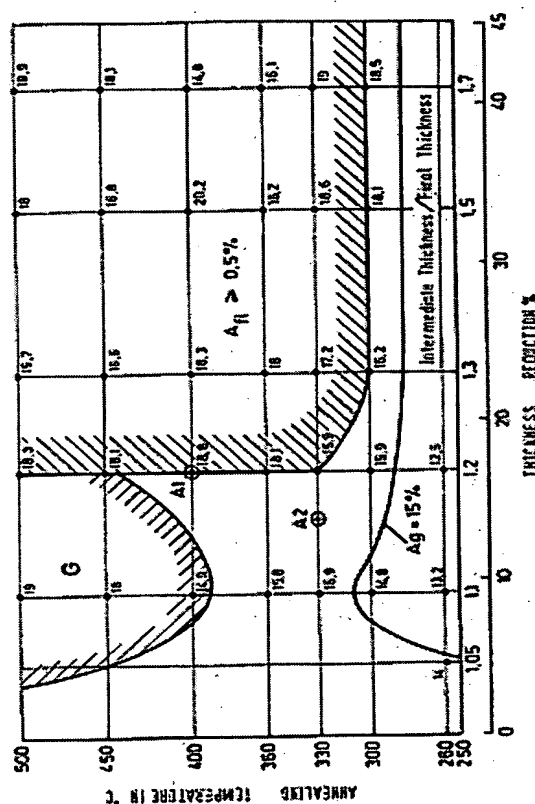


Fig.1

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(54) Method of manufacture of
 aluminium alloy sheets containing
 magnesium and zinc

(57) An Al-Mg alloy sheet with zinc
 addition in the range 0.5 to 2 wt.%,
 which is readily deformable in the soft
 condition, and especially suited for
 shaping into motor vehicle body
 components, has a strength of at least
 250 N/mm², a grain size of less than
 50 μ m, and after deformation has
 occurred, remains free of surface
 defects such as orange peel effect and
 Luders bands, and also remains
 insensitive to stress corrosion cracking

after subjection to heat.

The zinc addition produces a
 widening of the working range, so that
 after the last cold rolling one can
 always soft anneal above the
 recrystallisation temperature, without
 coarse grain or Luders bands
 appearing.

The zinc addition additionally
 produces an insensitivity to stress
 corrosion cracking, after a
 heterogenisation annealing
 subsequent to the soft annealing, even
 after a sensitisation at 150°C of the
 sheet which has been deformed with a
 cold reduction of 20% and more.

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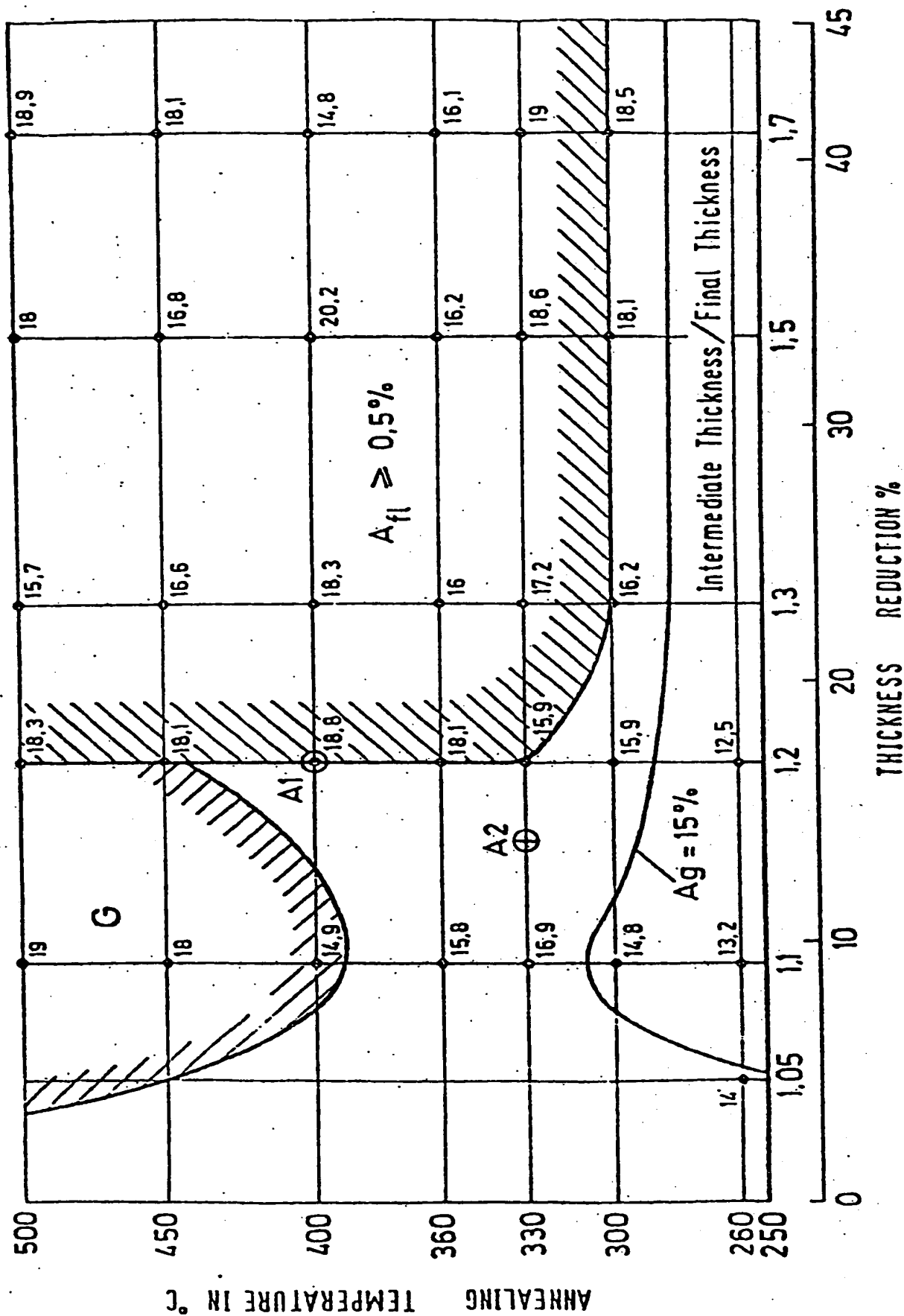


Fig.1

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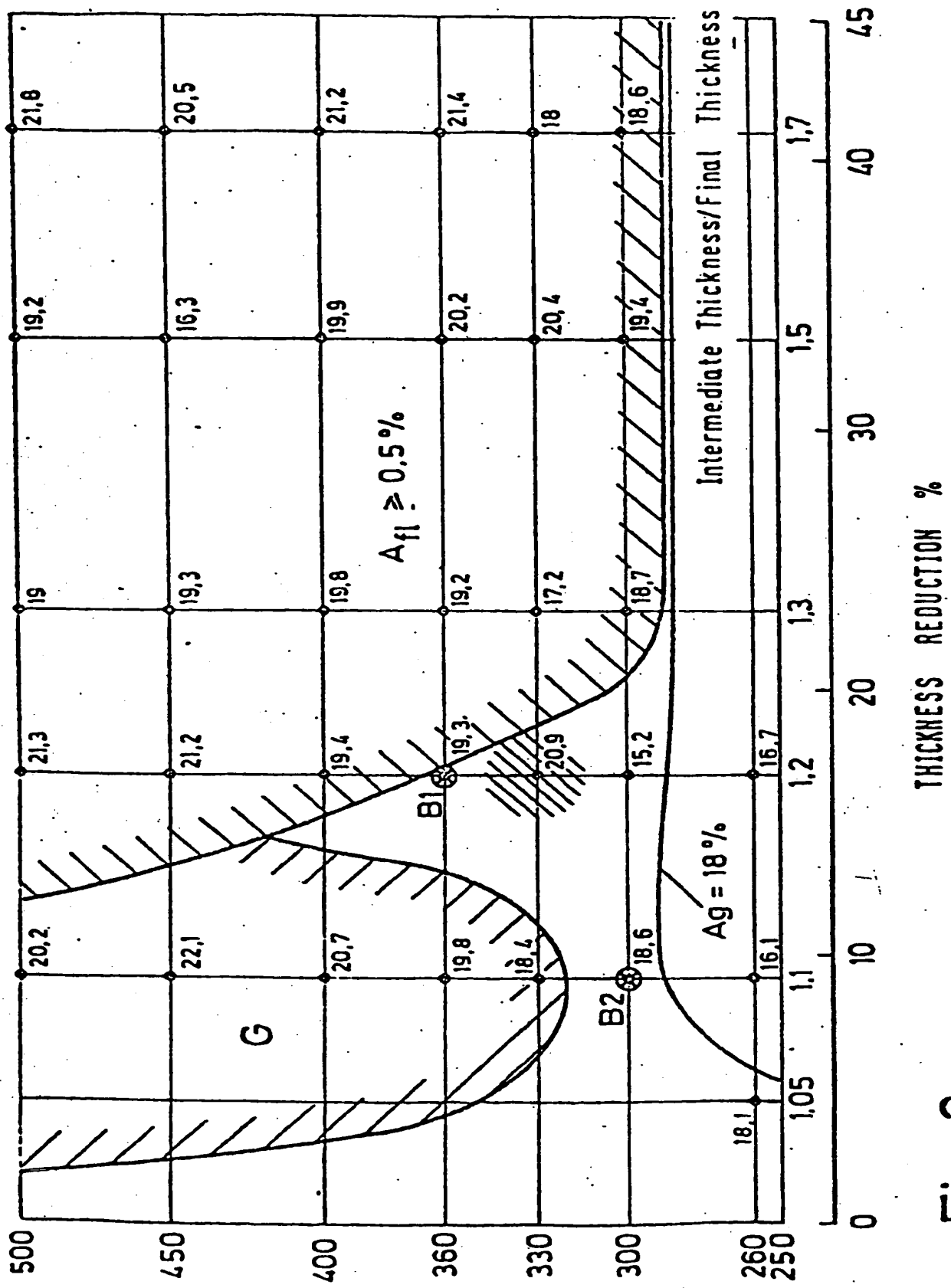


Fig. 2

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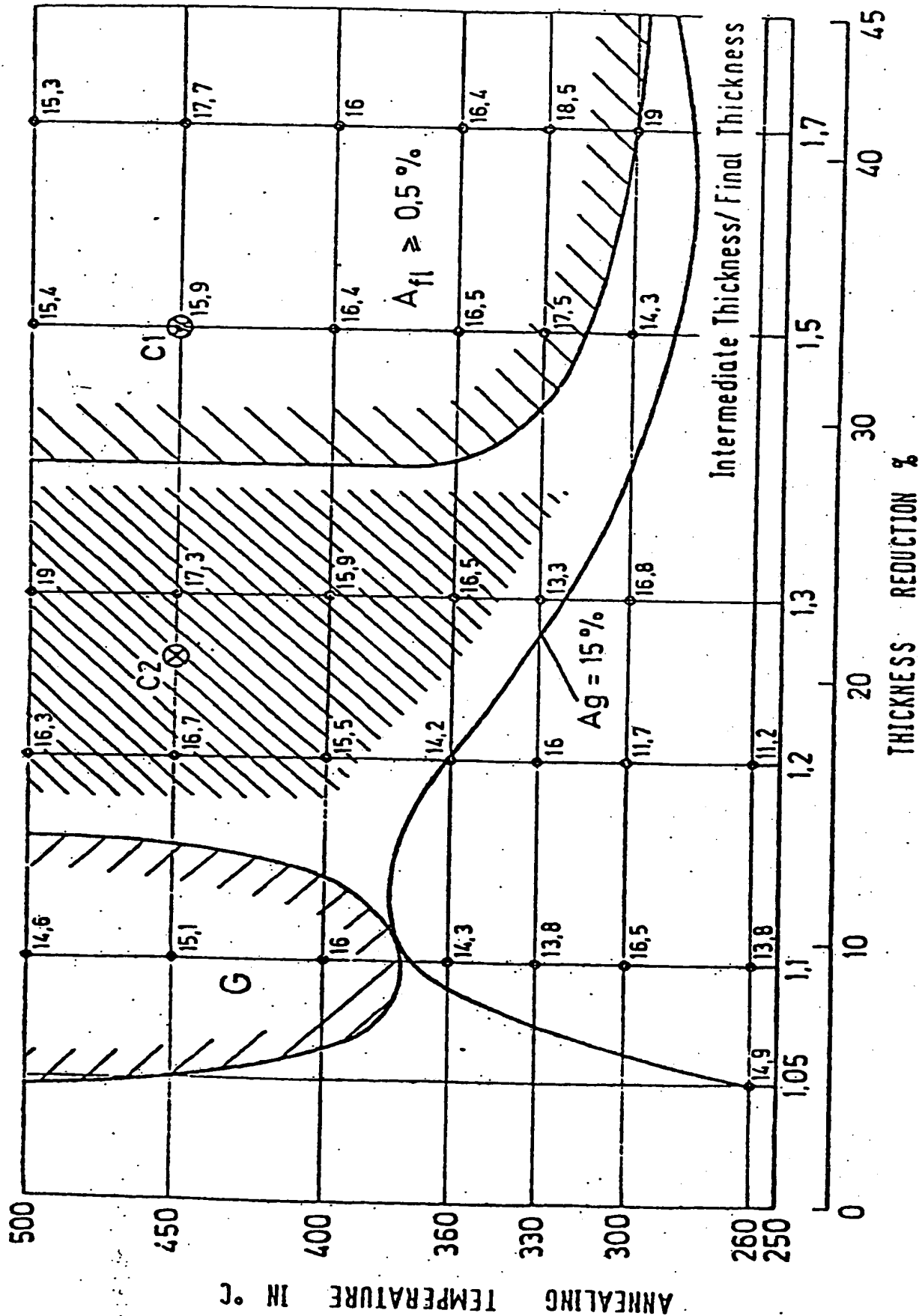


Fig. 3

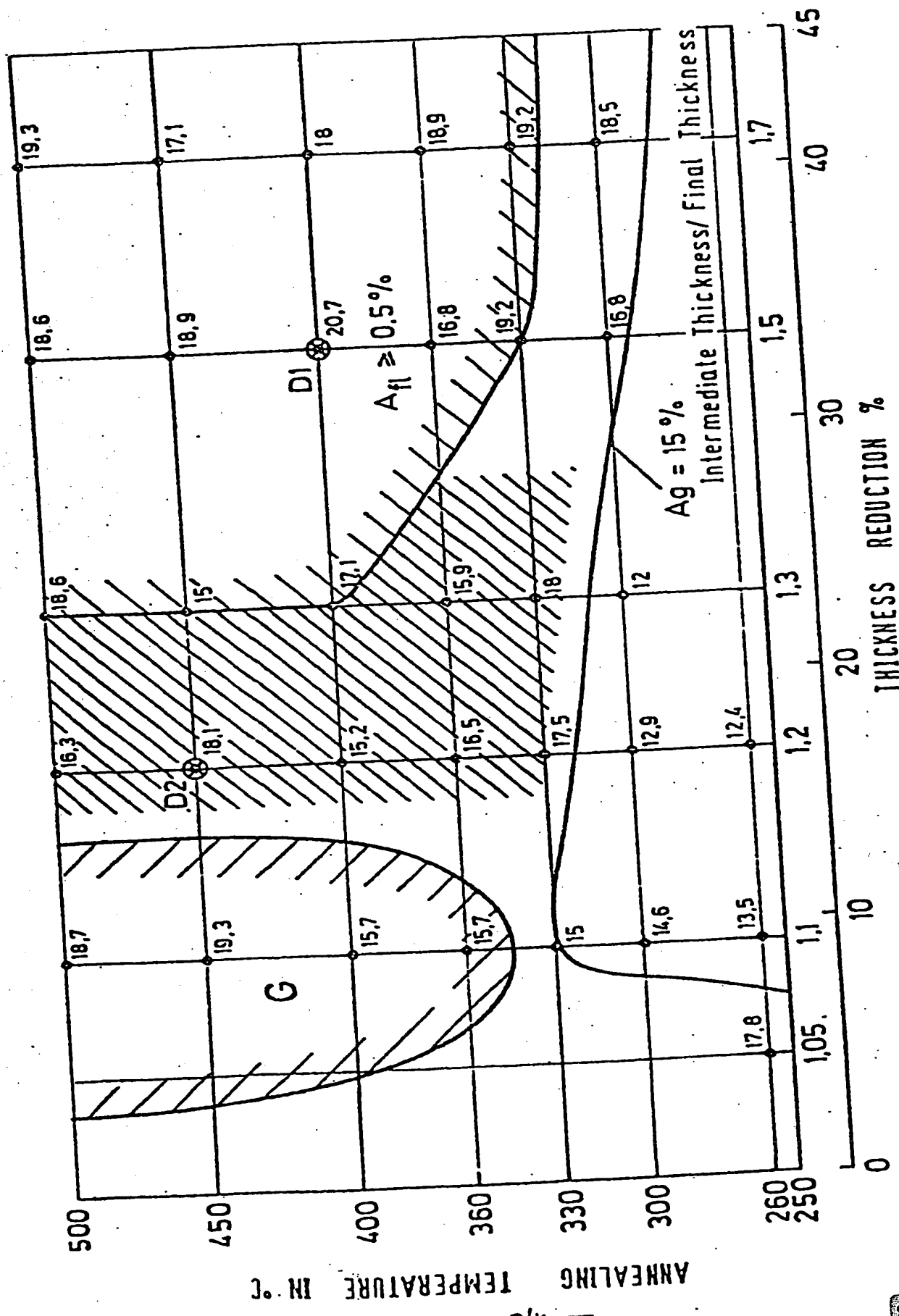
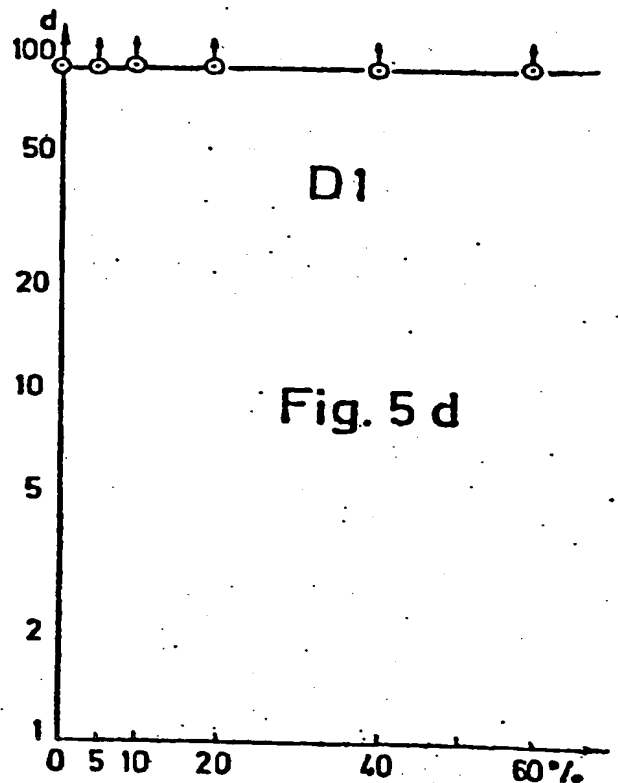
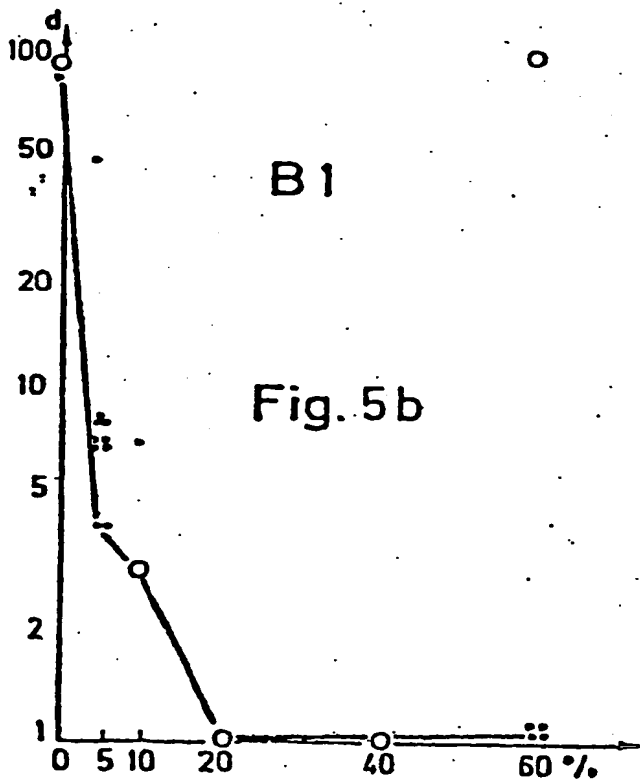
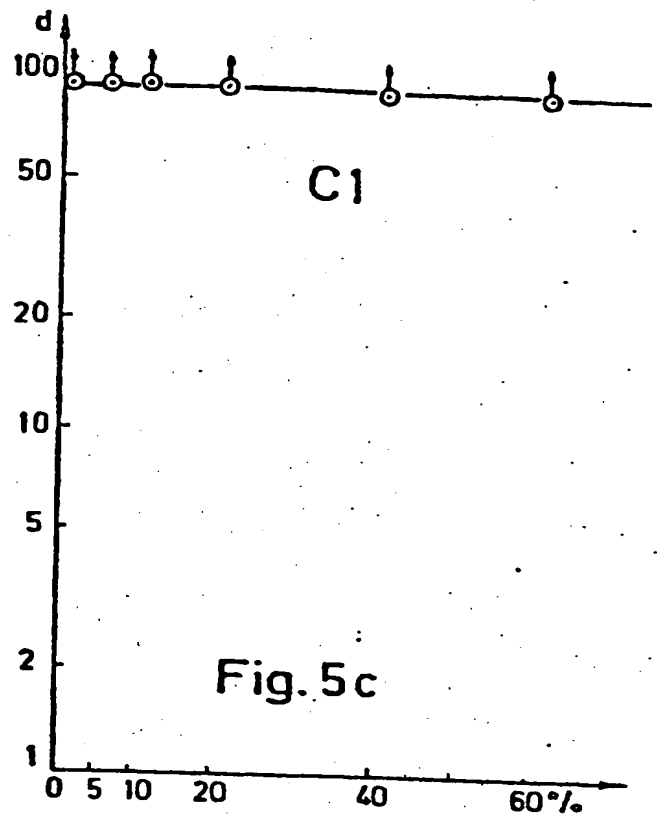
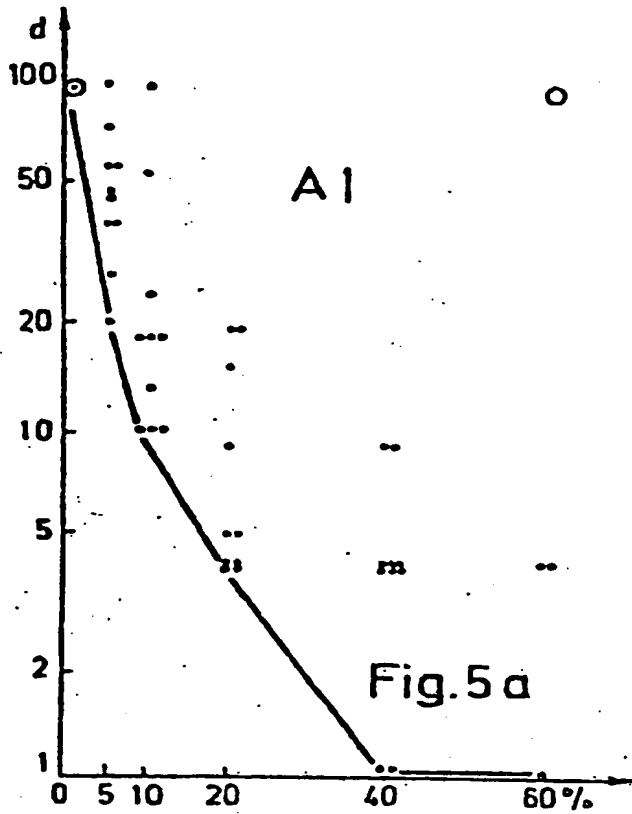


Fig. 4

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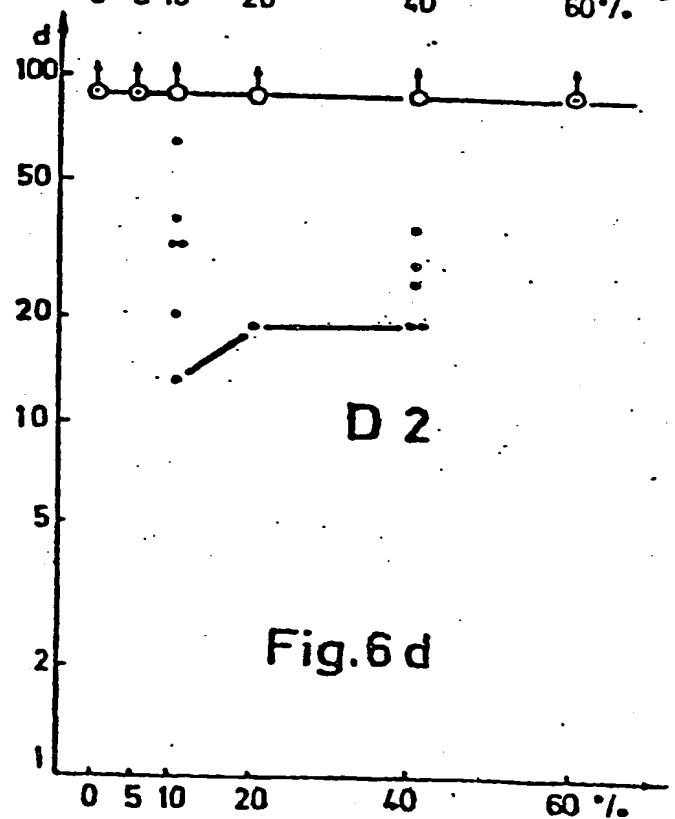
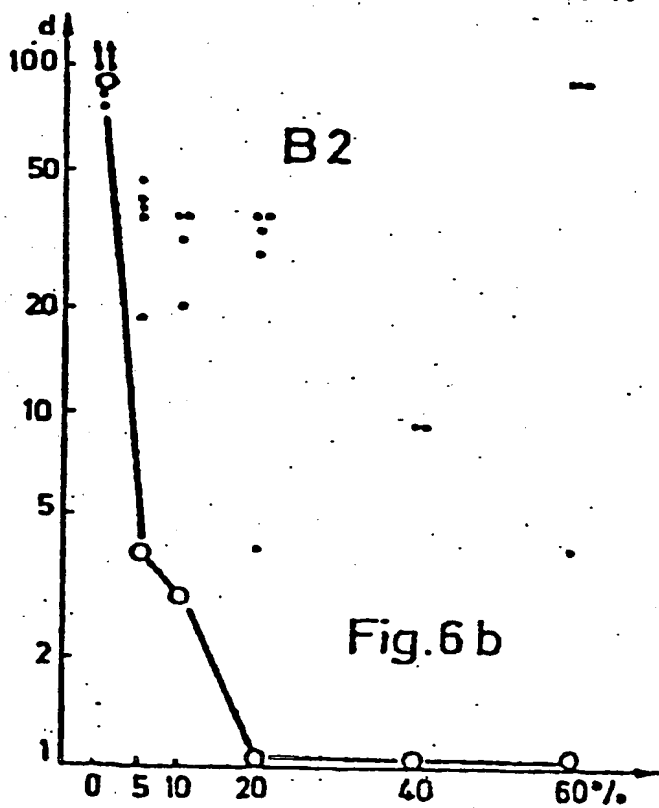
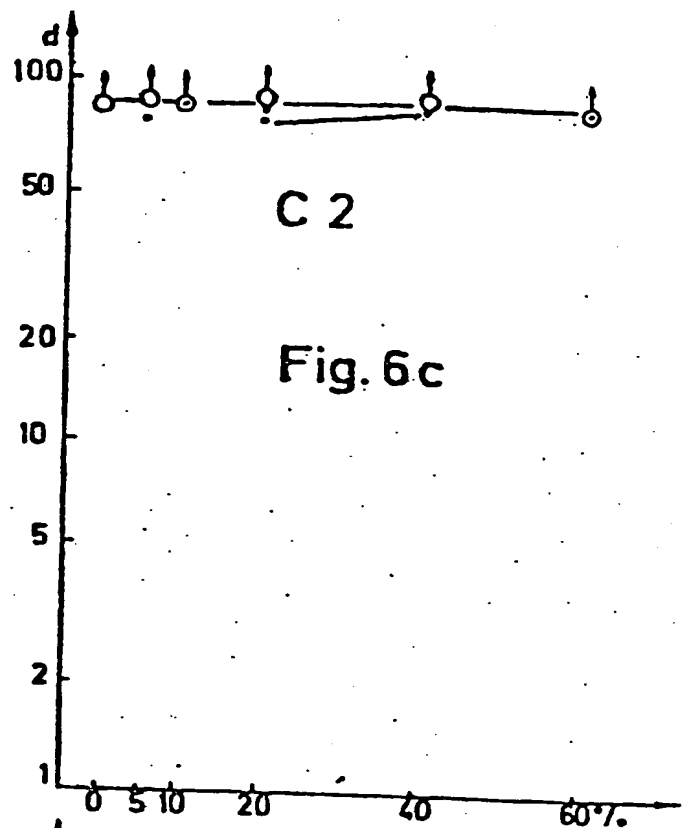
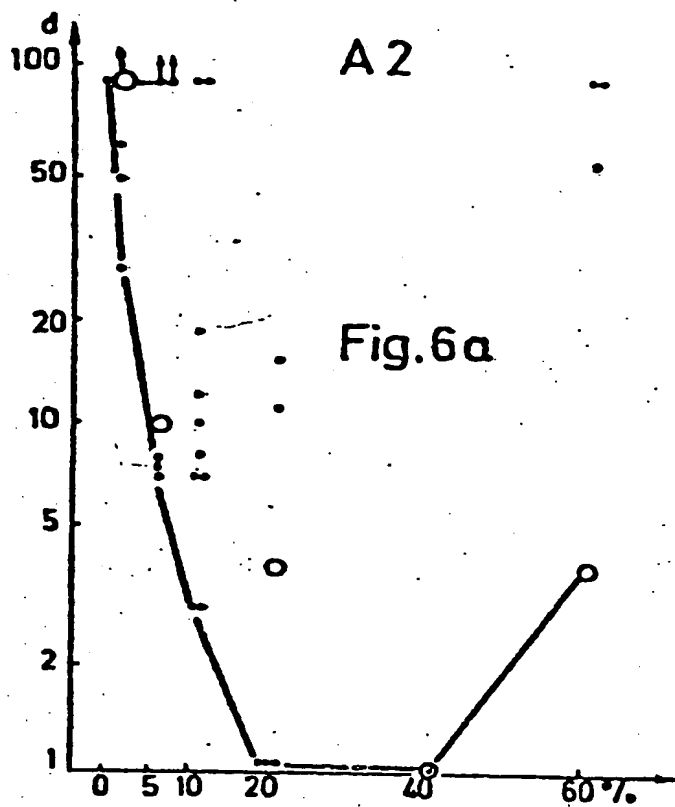
↑ NO CRACKS IN 90d

• SINGLE VALUES

○ ALL 10 VALUES

○ VALUES

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↑ NO CRACKS IN 90d
 ⊙ ALL 10 VALUES

○ REMAINING VALUES
 • SINGLE VALUES

SPECIFICATION

Method of manufacture of aluminum alloy sheets containing magnesium and zinc

Alloys with 4.5 to 5% magnesium together with additions of manganese and chromium attain, in the soft annealed condition, a tensile strength of 25 to 30 kg/mm².

However, alloys with high magnesium content display certain particularities, which must be taken account of during the manufacture and the further working by combined deep drawing and stretch forming of the preferred cold deformation in the manufacture of motor vehicle bodies.

Flow patterns of type A (corresponding to a marked plastic zone after passing beyond the yield point) cannot be tolerated in external components of bodies. Likewise after cold deformation and subsequent storage at elevated temperature the sheet must not be sensitive to stress corrosion cracking (SCC). Likewise undesired, because accompanied by a loss in strength and rigidity, is a partial disappearance of the strength condition produced during the cold deformation. Surprisingly, the stress corrosion cracking has not been taken account of hitherto in connection with use in vehicle bodies, where however all the conditions are present to produce stress corrosion cracking: the presence of deformation zones as well as of internal stresses from deep drawing or stretch forming, the usual subjection to high temperatures (waste heat of the engine, incident sunlight), as well as corrosive surroundings.

Treatments are known by which flow patterns can be avoided. These measures are however of such a nature that the latter are not suitable for a treatment of motor vehicle bodies. To these measures belong production of a grain diameter above 50 µm, which after cold deformation leads to a so-called orange peel effect on the surface of the cold-formed part — cold-deformation over the distinct flow zone of beyond about 1% remaining extension, which leads to a great loss in formability — and finally quenching from a soft annealing temperature in the solution range of about 530°C, which brings with it the disadvantage that, because of the only transient effect, the sheet must be immediately deformed, which makes storage practically impossible.

Likewise measure are known for reduction of sensitivity with respect to stress corrosion cracking, such as an addition of about 0.75% manganese and/or of about 1% zinc and finally a heterogenising annealing at 220 — 260°C, which has as consequence a precipitation at the grain boundaries like a string of pearls.

The combination of the measures for reduction of the SCC sensitivity with the above-mentioned measures for avoidance of flow patterns does not, even if it is possible, succeed in avoiding the disadvantages of the latter and in ensuring the resistance to SCC after a succeeding deformation.

With the invention a method is to be provided to optimise sheets of AlMg alloys according to the following criteria:

— Good formability with good resistance against stress corrosion cracking, freedom from flow patterns, and fine grain size.

The invention involves a method of manufacture of a sheet of an aluminium-magnesium alloy with a tensile strength of at least 250 N/mm² in the soft condition, particularly suited for the production of shaped components, which are free from orange peel effect and Luders bands, and, without special heat treatment after the deformation, are insensitive to a possible sensitisation in use with respect to stress corrosion cracking, characterised in that a rolling ingot is made from an aluminium-magnesium alloy with an addition of zinc in the range from 0.5 to 2 wt.%, that the working by various hot rolling, intermediate annealing, and cold rolling operations occurs in such a way that, after rolling to final thickness, a condition is present which, during soft annealing, leads to a grain size of less than 50 µm, and to a plastic extension in the marked plastic zone of 0—0.5% and that the sheet subsequent to the soft annealing is held for at least one hour in the temperature range from 220 to 226°C.

A preferred method is characterised by the following steps out in succession:

— production of a homogenised and surface-machined rolling ingot with a composition in weight percent of 4 to 7 Mg, max. 0.6 Si, max. 0.8 Fe, max. 1.0 Mn, max. 1.0 Cu, max. 0.3 Cr, 0.7 to 1.5 Zn, preferably 0.9 to 1.5 Zn, max. 0.05 Bi, balance essentially Al,

— hot rolling to intermediate thickness,

— cold rolling with a thickness reduction of at least 20%, preferably 30 to 70%.

— intermediate annealing above the recrystallisation temperature, preferably above 350°C,

— cold rolling to final thickness with a thickness reduction of at least 12%, preferably 15 to 30%.

— soft annealing above the recrystallisation temperature, preferably above 300°C,

— stabilisation against stress corrosion cracking by holding during 1 to 24 hours at temperatures between 200 and 260°C, preferably after cooling from the soft annealing temperature to the stabilisation temperature with a speed of less than 100°C per hour.

One can also cold roll directly to the final thickness from the intermediate thickness after hot rolling, and also the heterogenisation can be solely determined from the continuous cooling speed from the annealing temperature. In this kind of heterogenisation one can avoid holding in a temperature zone over a long period of time. Whether one or the other case, alone or in combination, is in question, is dependent on the thickness of the cast, homogenised and surface-machined hot rolling ingot, on the alloy employed, and not least on the particular manufacturing possibilities.

The zinc addition produces the advantage, hitherto not recognised of so widening the working range between coarse grain and flow patterns of type A, that fully sort sheet can be produced, which, in a subsequent cold deformation, shows neither orange pael effect nor flow patterns.

The method according to the invention, especially the effect of the addition of zinc, with the widened working range referred to through this addition of zinc, makes it possible for the first time to produce sheets for motor vehicle bodies, without having to fear that these, after cold working has occurred in the car factory, will fail by stress corrosion cracking. The method however is not limited to the manufacture of stock sheet for motor vehicle construction, but it is also suitable for preparation of stock sheet for similar cases.

In addition, by the method according to the invention a certainty is ensured that cannot be attained with zinc-free aluminium-magnesium alloys. This improvement in certainty facilitates stocking both by a semis manufacturer, and also by a manufacturer of bodies, and signifies an economical operation for the semis factory.

It has been found that the advantages mentioned do indeed appear in practice in use of the invention, and that fine-grained, high-strength sheets can be produced, which, even after deformation has occurred, retain a handsome surface (no Luders lines, no orange peel effect), and which moreover, even after exposure to heat, remain insensitive to stress corrosion cracking.

One particularly preferred range of composition for the rolling ingot is, in weight percent, 4.0 to 4.9 Mg, max. 0.4 Si, Max. 0.4 Fe, max. 0.1 Cu, 0.4 to 1.0 Mn, 0.05 to 0.25 Cr, 0.9 to 1.1 Zn, balance essentially Al.

Another particularly preferred range of composition for the rolling ingot is, in weight percent, 4.5 to 5.6 Mg, max. 0.4 Si, max. 0.5 Fe, max. 0.1 Cu, 0.1 to 0.6 Mn, max. 0.2 Cr, 0.9 to 1.1 Zn, balance essentially Al.

Preferably the material condition before the final annealing corresponds to a cold rolling degree of 15 to 22%.

Four versions A, B, C and D with the compositions set out in Table I were prepared and compared with one another.

TABLE I

Alloy constituent in wt. %	Alloy A	Alloy B	Alloy C	Alloy D
Silicon	0.15	0.15	0.15	0.15
Iron	0.25	0.22	0.22	0.22
Manganese	0.79	0.30	0.79	0.30
Magnesium	4.38	4.68	4.17	4.67
Zinc	—	—	0.97	1.00
Chromium	0.11	—	0.11	0.14
Bismuth	0.028	0.024	0.024	0.028
Aluminum x)	balance	balance	balance	balance

x) plus usual impurities dependent on the recovery process.

A corresponds to DIN reference AlMg4.5Mn or AA No. 5083, B corresponds to DIN reference AlMg5 or approximately AA No. 5056, the two zinc-containing alloys C and D are not standard ones.

Each of these alloys was cast into a rolling ingot 70 mm thick, and then homogenised at 480°C during 6 hours and 550°C during 12 hours, then surface machined and hot rolled in the usual manner to 4 mm.

Then followed a cold rolling to various thicknesses between 1 mm and 2 mm, which signified a reduction from the starting thickness of 75% to 50%. Then said cold rolled test pieces were annealed at 400°C, during which the sheets recrystallised with a fine grain. Thereafter all test pieces were cold rolled to a final thickness of 1 mm, with cold rolling degrees (percentage reduction of thickness) of 50%. The finally-rolled test pieces were annealed at 200 to 500°C, during which, according to the

degree of cold rolling and the temperature, a recovery or a partial or complete recrystallisation could occur.

Figures 1 to 4 show the outcome for the individual alloys A, B, C and D, and in them are noted, each in dependence of the annealing temperature and the reduction in thickness during cold rolling, the corresponding values of the uniform elongation A_{u} , as well as the coarse grain zone (G), and the zone where flow patterns-type A (Luders lines) occur ($A_{\text{u}} > 0.5\%$).

The uniform elongation serves as a measure of the formability during stretch forming or deep drawing. It was reckoned from elongation values A_{10} and A_2 provided in tensile tests according to the Kostron formula (H. Kostron "Zur Mathematik des Zugversuches," Archiv für das Eisenhüttenwesen, 22, 1951, page 317 et seq.).

The yield-to tensile ratio $RO.2/R_m$ was also taken into account as a measure of formability where, opposite from the case with the uniform elongation, lower values of $RO.2/R_m$ enable one to recognise a greater formability during deep drawing/stretch forming. Additional information is given over the degree of softening by the annealing.

TABLE II

Alloy	Annealing temperature °C	Intermediate thickness mm	ϵ %	R _m kp/mm ²	R _{0.2} kp/mm ²	R _{0.2} /R _m %	A _g %	A _{II} %	Grain size μ m
A	330	1.1	9	32.9	18.5	56	16.9	0	not
	360	1.1	9	32.8	18.1	55	15.8	0	recrystallised
	300	1.2	16	31.0	14.5	47	15.9	0.3	
B	260	1.2	16	30.2	18.6	62	16.7	0	not
	300	1.2	16	27.2	11.3	42	15.8	0.5	recrystallised
	330	1.2	16	27.2	11.2	42	20.9	0.4	38
C	330	1.2	16	30.0	11.9	40	18.0	0.1	34
	360	1.2	16	30.2	11.9	40	14.2	0	35
	400	1.2	16	30.3	11.9	40	15.5	0	30
	450	1.2	16	30.2	11.9	40	16.7	0	29
	500	1.2	16	29.9	11.6	39	18.3	0	36
	330	1.3	23	30.1	12.1	40	13.3	0.4	24
	360	1.3	23	30.5	12.5	41	16.5	0.4	20
	400	1.3	23	30.4	12.6	41	15.9	0.4	26
	450	1.3	23	30.4	12.4	41	17.3	0.3	26
	500	1.3	23	30.1	12.2	41	19.0	0.4	26

TABLE 11 (Continued)

Alloy	Annealing temperature °C	Intermediate thickness mm	ϵ %	R _m kp/mm ²	R _{0.2}	R _{0.2} /R _m %	A _g %	A ₁₁ %	Grain size μ m
D	330	1.2	16	29.9	11.9	40	17.5	0	46
	360	1.2	18	30.1	11.9	40	16.5	0	40
	400	1.2	18	29.8	12.0	40	15.2	0.25	36
	450	1.2	18	29.6	11.9	40	18.1	0.3	38
	500	1.2	16	29.8	12.0	40	16.3	0.2	32
	330	1.3	23	30.3	12.6	42	18	0.3	30
	360	1.3	23	30.2	12.7	42	15.9	0.4	20
	400	1.3	23	30.0	12.6	42	17.1	0.5	26
	450	1.3	23	29.7	12.6	42	15	0.5	14
	500	1.3	23	29.6	12.6	42	16.6	0.5	29

Notes: ϵ = Thickness reduction from intermediate thickness to 1 mm final thickness.

R_m = Tensile strength.

R_{0.2} = Elastic limit

R_{0.2}/R_m = Yield to tensile ratio.

A_g = Uniform elongation.

A₁₁ = Extension in marked plastic zone.

In the drawings, the working ranges for the alloys A-B-C-D were indicated. These working ranges lie within an area which is delimited by the boundaries of coarse grain structure and of flow patterns type A, as well as by the contour line for the uniform elongation A_g of about 15%. Beyond this, the sheet must be totally recrystallised.

5 Since the tensile strength with all alloys and all shown combinations of thickness reduction and annealing temperature has remained above 27 kp/mm² (270 N per mm²), a tabular rendering of the total experimental area is omitted. 5

10 In Table II the individual values are collected from the tensile test, which are in question for the working range, in dependence upon the annealing temperature (annealing period 1 h), cold rolling degree and grain size. Among other things, there results the expected correlation between the yield to tensile ratio $RO.2/R_m$ and the degree of softening. In accordance with it, the values of $RO.2/R_m$ of 39 to 42% were associated with the grain sizes 14 to 40 μm (C and D), while with values of $RO.2/R_m$ of 47 to 62% no recrystallisation had yet occurred (A and B). With A therefore there resulted no working range and with B only one lying around a single point. 10

15 As coarse grain is understood a mean grain diameter of more than 50 μm . As a measure for the working range of each alloy there can serve the area in cm² of the zone indicated with shading in Figures 1 to 4 according to the named criteria. 15

— The result of the alloy A is an area of zero cm², for the alloy B an area of about 2.1 cm², for the alloy C an area of about 43 cm², for the alloy D an area of about 43 cm².

20 The zinc addition in the alloys C and D thus according to the above results brings a significant and hitherto unknown broadening of the working range with it, because the flow patterns first appear with smaller grain sizes. 20

Before all else, with these alloys the annealing treatment can be selected so high that it always gives rise to a complete recrystallisation of the cold-rolled sheet.

25 In Figures 1 to 4, the combinations of reduction in thickness and annealing temperature, which were chosen for the testing of the behaviour in stress corrosion cracking, are indicated as A1/A2, B1/B2, C1/C2, and D1/D2. The annealing period amounted universally to one hour. 25

The versions A1, A2, B1 and B2 are state of the art, which are to be compared with the versions according to the invention C2 and D2 as regards behaviour in stress corrosion cracking before and after a new cold deformation has occurred. 30

The versions C1 and D1 fall much within the area of flow patterns, which is characterised by plastic extensions in the marked flow zone of more than 0.5%. These sheets can be employed if it does not so much matter whether flow patterns occur or not. These sheets can be employed for the internal construction of a motor vehicle or the like. 30

35 The following Table III represents the starting parameters of the versions indicated in Figures 1 to 4. 35

The marked flow zone corresponds in the versions A1 — D1 to plastic extensions of 0.5 — 0.7%, in the versions A2 — D2 those of 0 — 0.5%.

40 The behaviour in stress corrosion cracking was tested by means of U-bend-specimens in accordance with DIN 50908/1964 up to a duration of 90 days. For the U-bend tests, the soft annealed or weakened and heterogenised sheets of versions A1 to D1 and A2 to D2 were cold rolled with thickness reductions of 0% to 60%, and then subjected for 3 days to a temperature of 150°C, to make these sensitive with respect to stress corrosion cracking (sensitisation). 40

TABLE III

Version	R _m kp/mm ²	R _p kp/mm ²	R _p /R _m %	A _g %	A _{II} %	Grain size μm	Criteria met?
A1	30.7	13.1	42.6	18.8	0.6	< 50	no, not free of flow patterns
A2	30.5	13.2	43.3	15.9	0.5	no recryst.	no, no recrystallisation
B1	27.4	11.4	41.6	19.3	0.7	35	no, not free of flow patterns
B2	28.9	15.2	55.0	18.6	0	not recryst.	no, no recrystallisation
C1	30.6	13.7	44.7	15.9	0.6	25	no, not free of flow patterns
C2	30.1	12.1	40.2	16.3	0.2	28	yes
D1	30.2	13.6	45	20.7	0.7	24	no, not free of flow patterns
D2	29.6	11.9	40.2	18.1	0.3	38	yes

R_m = Tensile strengthR_p = Elastic limitR_p/R_m = Yield to tensile ratioA_g = Uniform elongationA_{II} = Extension in marked plastic zone

The testing solution consisted of:

30 g NaCl

5.44 g $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$

5.68 g $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$

balance de-ionised water to 1 litre solution,

appr. 7.5 ml acetic acid (>98%) added, to stabilise the solution at a pH of 4.

Testing temperature was 25°C.

For the versions A1, B1, C1 and D1, which were heterogenised at 220°C during 8 hours, the results are shown graphically in Figures 5a, 5b, 5c and 5d. An analogous showing is given in Figures 6a — 6d for the versions A2, B2, C2 and D2. In these versions, the heterogenisation consisted simply of a slow cooling from the annealing temperature (from 400°C within about 4 hours to 250°C).

For understanding of Figures 5 and 6, the explanation of Figure 5a is sufficient, which relates to version A1. For rolling degrees of 0%, 5%, 10%, 20%, 40% and 60%, the life in days is shown of 10

In order to be able to compare the versions C2 and D2, or C1 and D1 better with the versions A2 and B2, or A1 and D1, which represent the state of the art, in Figures 5 or 6 corresponding diagrams are arranged adjacent to each other, and the points with the lowest life are connected by straight lines if possible.

Whereas with the zinc-free alloys A and B such a polygon can be drawn always, and independently of the degree of cold deformation, this is not possible with the zinc-containing versions C1 and D1, and with the version C2 one obtains only a single straight line between 20 and 40%, with the version D2 the polygon first begins at all at 10%, and ceases already at 40%.

Even the points of such tests, which showed no cracks even after lapse of 90 days (indicated with an arrow) were when possible likewise connected together with straight lines. With the zinc-containing alloys C and D it was always possible to run a straight line through all the points with a life of over 90 days, quite independently of the degree of deformation, whereas with the zinc-free versions A1 and B1 this is in general not possible, and with A2 and B2 can only be done with degrees of deformation between 0 and 5%.

Despite incomplete heterogenisation, the versions according to the invention C2 and D2 are essentially less sensitive with respect to SCC than the zinc-free comparison versions A2 and B2.

The results establish that, by a heterogenisation in addition to the soft or weakening annealing, one can succeed in making sheets of AlMg4.5Mn or AlMg5, in the condition in which they had left the pre-form factory, more or less insensitive with respect to stress corrosion cracking. With the versions A1 and B1, which were heterogenised at 220°C for 8 hours, this was achieved better than with the versions A2 and B2, which only slowly cooled to 250°C after the softening or recovery annealing.

However, after a cold deformation of more than 20%, the sheets of AlMg4.5Mn and AlMg5 again become sensitive to stress corrosion cracking, if they are subjected to long heating to moderately high temperatures. Cold deformation in this zone can occur during the production of motor vehicle bodies with combined stretch forming and deep drawing.

If, however, the heterogenisation treatment is undertaken on an AlMg alloy zinc addition, then the sheet even remains insensitive with respect to stress corrosion cracking, in cases in which cold deformation is undertaken before the critical heat influence (sensitisation). The bodies of motor vehicles of sheets of zinc-containing AlMg alloys, which had been produced by the manufacturing method according to the invention, bring to the manufacturer and purchaser of motor vehicles no disadvantageous trouble as regards cracks, which have arisen through stress corrosion cracking. A further advantage arises for the manufacturer of motor vehicles from the fact that prepared body work components can be stored even without surface protection.

The heterogenisation annealing after the last soft annealing produces with zinc-containing AlMg alloys finely dispersed precipitation of MgZn phases even in the grain interiors, while the heterogenisation annealing with zinc-free AlMg alloys produces precipitations or AlMg phases only in the grain boundaries, so that in deformation bands which arise during subsequent deformation while under the influence of elevated temperatures, associated precipitations can occur freshly, which lead to stress corrosion cracking.

CLAIMS

1. Method of manufacture of a sheet of an aluminium-magnesium alloy with a tensile strength of at least 250 N/mm² in the soft condition, particularly suited for the production of shaped components, which are free from orange peel effect and Luders bands, and, without special heat treatment after the deformation, are insensitive to a possible sensitisation in use with respect to stress corrosion cracking, characterised in that a rolling ingot is made from an aluminium-magnesium alloy with an addition of zinc in the range from 0.5 to 2 wt.%, that the working by various hot rolling, intermediate annealing and cold rolling operations occurs in such a way that, after rolling to final thickness, a condition is present which, during soft annealing, leads to a grain size of less than 50µm, and to a plastic extension in the marked plastic zone of 0—0.5% and that the sheet subsequent to the soft annealing is held for at least one hour in the temperature range from 220 to 260°C.

2. Method according to claim 1, characterised by the following steps carried out in succession:
- production of a homogenised and surface-machined rolling ingot with a composition in weight percent of 4 to 7 Mg, max. 0.6 Si, max. 0.8 Fe, max. 1.0 Mn max. 1.0 Cu, max. 0.3 Cr, 0.7 to 1.5 Zn, preferably 0.9 to 1.5 Zn, max. 0.05 Si, balance essentially Al,
 - 5 — hot rolling to intermediate thickness,
 - cold rolling with a thickness reduction of at least 20%, preferably 30 to 70%,
 - intermediate annealing above the recrystallisation temperature, preferably above 350°C,
 - cold rolling to final thickness with a thickness reduction of at least 12%, preferably 15 to 30%,
 - soft annealing above the recrystallisation temperature preferably above 300°C,
 - 10 — stabilisation against stress corrosion cracking by holding during 1 to 24 hours at temperatures between 200 and 260°C, preferably after cooling from the soft annealing temperature to the stabilisation temperature with a speed of less than 100°C per hour.
3. Method according to claims 1 and 2, characterised by an alloy with a composition in weight percent of 4.0 to 4.9 Mg, max. 0.4 Si, max. 0.4 Fe, max. 0.1 Cu, 0.4 to 1.0 Mn, 0.05 to 0.25 Cr, 0.9 to 1.1 Zn, balance essentially Al for the rolling ingot.
- 15 4. Method according to claims 1 and 2, characterised by a composition in weight percent of 4.5 to 5.6 Mg, max. 0.4 Si, max. 0.5 Fe, max. 0.1 Cu, 0.1 to 0.6 Mn, max. 0.2 Cr, 0.9 to 1.1 Zn, balance essentially Al for the rolling ingot.
5. Method according to claim 4, characterised in that the material condition before the final 20 annealing corresponds to a cold rolling degree of 15 to 22%.
6. Method according to claims 1 to 5, characterised in that the said alloy sheets are employed for production of motor vehicle body components.

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